

TITLE

AQUEOUS THIXOTROPES FOR WATERBORNE SYSTEMS

BACKGROUND OF THE INVENTION

1. Field of Invention:

5 The present invention relates to aqueous thixotropes for waterborne systems and, more particularly, to aqueous dispersions of fumed silica for use as effective thixotropes and rheology control agents in waterborne systems.

2. Description of the Related Art:

Both hydrophilic and hydrophobic fumed silicas are widely used in the coating industry 10 to improve rheology, for flow control and storage stability, as well as serve as an anti-settling agent for pigments and fillers. The production of hydrophilic fumed silica is a well known process. Grades vary in particle and aggregate size. Hydrophobic silica can be produced by treating a fumed silica with a suitable agent which will vary depending on the desired degree of hydrophobicity and other characteristics. Such treating agents include, for example, polydimethylsiloxane oils of various molecular weights, dimethylchlorosilane, trimethoxyoctylsilane, disilazanes, such as hexamethyldisilazane (HMDZ), and mixtures thereof.

As environmental awareness increases, manufacturers face increased pressure to replace conventional solvent based systems. As a result, aqueous systems are increasingly used in many applications such as automotive and industrial coatings, paints, inks, adhesives, and the like. 15 While hydrophilic and hydrophobic silicas have both been used in solvent-based coating formulations on a commercial scale, their use in aqueous formulations have been plagued with disadvantages. For example, in aqueous systems, either the silica additive must be increased to unacceptable levels or the formulation does not attain the desired level of performance. A demand, therefore, exists for aqueous systems which perform comparably to solvent based systems and, accordingly, for improved additives or methods to accomplish such results.

It is therefore an object of the present invention to provide a versatile and efficient rheological additive for waterborne systems, such as in coatings and other industrial applications. A further object is to provide an additive that fosters stability in aqueous compositions. A still further object is to provide a thixotrope which alleviates many of the waterborne coating rheology 20 control formulating difficulties that exist with other inorganic and organic materials.

SUMMARY OF THE INVENTION

In aqueous systems, which include both emulsions and water reducible vehicles, untreated (hydrophilic) silicas are typically not effective because of the large concentrations of silica that are necessary to realize the desired thickening. However, methods exist for increasing the thickening capability of a given concentration of silica by using certain substances as additives to modify the nature of the system. For example, in systems that are not readily responsive to fumed silica because of inherent chemical properties, the correct additive can often facilitate efficient viscosity and thixotropic control. For example, in highly hydrogen-bonding liquids, the additives that are typically most useful in improving the thickening and thixotropic efficiency of fumed silica are cationic surfactants. The surfactants modify and partially impede the interaction between fumed silica and the solvent, thereby allowing the fumed silica network structure to

Accordingly, the present invention is directed to an aqueous dispersion of fumed silica that provides enhanced rheology control and thixotropy to waterborne systems. The fumed silica dispersion is uniformly dispersed in the waterborne system such that an amount ranging between 25 0.5% and 10.0%, by weight, of silica is present in the final composition. The waterborne system, therefore, includes an aqueous dispersion of fumed silica, and a waterborne resin. The resin is selected from the group consisting of alkyd, acrylic, polyester, polyether, silicate, urethane, epoxy, formaldehyde, vinyl, and mixtures thereof. The fumed silica has a surface area between about 85 m²/g and about 410 m²/g.

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BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a graph of the rheological performance of an epoxy resin system incorporating the present fumed silica dispersion;

FIG. 2 is a graph of the rheological performance of an bisphenol-A epoxy resin system incorporating the present fumed silica dispersion;

FIG. 3 is a graph of the rheological performance of an epichlorohydrin and bisphenol-A resin system incorporating the present fumed silica dispersion;

FIG. 4 is a graph of the rheological performance of a urethane modified epoxy resin system incorporating the present fumed silica dispersion; and

FIG. 5 is a graph of the rheological performance of an acrylic resin system incorporating the present fumed silica dispersion; and

FIG. 6 is a graph of the response of the present fumed silica dispersion in a typical water reducing alkyd coating formulation.

DETAILED DESCRIPTION OF THE INVENTION

15. The present invention is directed to an aqueous dispersion of hydrophilic fumed silica which provides enhanced rheology control and thixotropy to aqueous or waterborne systems. The present aqueous dispersion of fumed silica is effective in alkyl, acrylic, polyester, Polyether, silicate, urethane, epoxy, formaldehyde, vinyl, and mixtures or modifications thereof as vehicle systems. It has been found that unlike other organic and inorganic materials used as thixotropes 20 in waterborne systems such as coatings, the present aqueous dispersion of fumed silica will not typically react with additives in the formulation to produce unexpected side effects after product manufacture.

Fumed silicas useful in this invention are generally characterized by a chain-like structure having high surface area per unit weight. The production of fumed silica is a well-documented process which involves the hydrolysis of silicon tetrachloride vapor in a flame of hydrogen and oxygen. Molten particles of roughly spherical shapes are formed in the combustion process, the diameters of which are varied through process parameters. These molten spheres of fumed silica, 25 typically referred to as primary particles, fuse with one another by undergoing collisions at their contact points to form branched, three dimensional chain-like aggregates. The force necessary

to break aggregates is considerable and often considered irreversible because of the fusion. During cooling and collecting, the aggregates undergo further collision that may result in some mechanical entanglement to form agglomerates. Compared to the aggregates where the primary particles are fused together, agglomerates are thought to be loosely held together by Van der Waals forces and can be reversed, i.e. de-agglomerated, by proper dispersion in suitable media.

5. The size of the primary spherical particles that comprise the fumed silica aggregates determine the surface area. The surface area of the fumed silica, as measured by the nitrogen adsorption method of S. Brunauer, P. H. Emmet, and J. Teller, J. Am. Chemical Society, Volume 60, Page 309 (1938) and commonly referred to as BET, typically ranges from about 85 m²/g to 10 about 410 m²/g. In the present invention, the fumed silica is preferably in a range from about 175 m²/g to about 225 m²/g, and are of a high purity. High purity means that the total impurity content is typically less than 1% and preferably less than 0.01% (i.e., 100 ppm). Although many commercially available fumed silicas are suitable, CAB-O-SIL® fumed silica, available from the Cab-O-Sil Division of Cabot Corporation, Tuscola, IL, having a surface area of about 200 m²/g 15 is most preferred. Such a silica has been found to be of high quality and is readily dispersable.

The fumed silica of the present invention is uniformly dispersed in a stable aqueous medium (e.g. deionized water) using conventional methods known to those skilled in the art. By uniformly dispersed is meant that the aggregates are isolated and well distributed throughout the medium. By stable is typically meant that the aggregates will not re-agglomerate and settle out 20 (e.g. form a hard, dense sediment). The fumed silica dispersion should have a pH between 5 and 10.5 and may be adjusted by the addition of a suitable base such as sodium hydroxide, potassium hydroxide, ammonia and the like. Preferably, the fumed silica dispersion of the present invention has a pH ranging between 7.0 and 9.5. The fumed silica dispersion of the present invention is preferably prepared by the method described by Müller et al., in U.S. Patent

25. No. 5,246,624, the disclosure of which is incorporated herein in its entirety by reference. Although many commercially available fumed silica dispersions are suitable, CAB-O-SPERSE® aqueous fumed silica dispersions, available from the Cab-O-Sil Division of Cabot Corporation, Tuscola, IL, are most preferred.

The waterborne systems of the present invention are prepared by combining or mixing the 30 aqueous dispersions of fumed silica directly with a waterborne resin, such as an alkyd, acrylic,

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polyester, silicate, urethane, epoxy, and the like, or with a formulation containing a waterborne resin under low shear conditions (i.e., to prevent foaming) until a uniform homogeneous composition is obtained. Typically, the fumed silica dispersions range from about 10% to about 45% solids, by weight; and, preferably, between 15% and 30% solids, by weight. Most preferably, a fumed silica dispersion of about 20% solids, by weight, has been found to maximize the loading level of silica while maintaining the colloidal stability of the dispersion.

The aqueous dispersions of fumed silica of the present invention are useful in aqueous systems to provide effective rheological control (i.e., viscosity and thixotropy) for example, in industrial and automotive coating, adhesive, paint, and ink applications. It has been found that another advantage of using an aqueous dispersion of fumed silica in waterborne resin systems is to provide rheology and sag control, and anti-settling. It is also believed that a stable matrix is formed in waterborne formulations after the incorporation of the fumed silica dispersion due to electrostatic interaction. During the high-shear processes usually found in product application, the matrix disintegrates, thereby reducing viscosity and permitting near-Newtonian flow. After application, the inorganic matrix reforms at a predictable rate to provide resistance to sagging and edge-pull during film coalescence and/or cure. This matrix remains unmodified through the many physical film changes during conversion from liquid to solid, thus providing a predictable application consistency.

10 The present aqueous dispersions of fumed silica used as aqueous thixotropes in water-based systems are responsive to changes in pH, but have been found to be effective in the 7.0 to 9.5 pH range commonly used in products formulated for industrial and commercial use. The present dispersions have also been found to be effective in higher pH ranges, depending specifically upon the individual formulation.

15 Although the loading level, as a percent of total resin solids, the precise method of incorporation, and the stage of manufacture at which this is accomplished, all play a significant role in the final effectiveness of the dispersion, the aqueous dispersion of fumed silica of the present invention has been shown to be effective in many systems used in formulating waterborne products.

The present invention will be further illustrated by the following examples, which are intended to be illustrative in nature and are not to be considered as limiting the scope of the invention.

EXAMPLE I

The aqueous dispersion of fumed silica has been found to be an effective suspension agent in some formulations due to its internally-generated structure. The product forms a matrix capable of stopping or significantly retarding undesirable striation and pigment settling, even in products containing powdered zinc or other unusually heavy pigments. It is noted that the addition of dry hydrophilic fumed silica to waterborne systems has been found to be ineffective as a rheology control agent. In particular, the viscosity of the system will tend to continually increase over time, thereby not achieving stability. In addition, the dry silica is difficult to handle and disperse, and may tend to settle out at higher loading levels.

An aqueous colloidal dispersion of fumed silica, which can be used as an aqueous thixotrope in the present invention, was prepared and evaluated as follows.

20 Approximately 205.24 grams of deionized water and 0.4 gram of 38% hydrochloric acid were added to a commercial Waring blender. While mixing, 80 grams of CAB-O-SIL® PTG grade fumed silica were added to the blender. When the addition was complete, the mixture was stirred at a high rate for about five minutes. After the stirring was completed, about 108.52 grams of water were added to the mixture followed by about 6.12 grams of a 10% potassium hydroxide solution stabilizer. After the addition of the stabilizer, the mixture was stirred for an additional two minutes. The aqueous fumed silica dispersion prepared had a 20% solids level, a viscosity of about 65 centipoise, and a pH of 9.

Other advantages of the present dispersions are that they will not migrate in wet or dry films and are unaffected by heat and atmospheric exposure in the dried/cured film. The dispersion is inherently non-yellowing and will not contribute to color changes or drift in either

the wet or dry state. Moreover, the present aqueous dispersions of fumed silica are biologically inactive and is not expected support microbial activity.

EXAMPLE II

A scaled-up volume of the dispersion of EXAMPLE I was prepared, using 2-amino-2-methyl-1-propanol, available as AMP-95™ from Angus Chemical Company, Buffalo Grove, IL, as the base-stabilizer. Approximately 1283.80 grams of water were mixed with 0.251 gram of 5 38% hydrochloric acid. About 499.92 grams of CAB-O-SIL® PTG grade fumed silica were then added to the water/acid mixture, and was stirred for about 20 minutes. 678 grams of deionized water was then added to the mixture, followed by 10.54 grams of the AMP-95™ stabilizer. The aqueous fumed silica dispersion prepared had a 20.22% solids level, and a pH of 9.

EXAMPLE III

10 A waterborne system, including an aqueous fumed silica dispersion (similar to the dispersion prepared in EXAMPLE I) and a waterborne epoxy resin, was prepared and evaluated to determine the effectiveness of the present dispersion as a thixotrope for waterborne systems. The aqueous fumed silica dispersion was mixed with Waterpoxy® 701 Resin epoxy curing agent, available from Henkel Corporation, Ambler, PA, until a loading level of 2% dry silica on resin 15 solids was achieved. The viscosity (centipoise), shear thinning index (STI), and pH were then measured after sample preparation (0 days), and after periods of 1 and 7 days. The viscosity was measured on a Brookfield viscometer using Nos. 3 or 4 spindle at 6 and 60 RPM's. The experimental results are presented below in Table 1.

TABLE I

20	25	20	25	20	25
VISCOSITY	DAYS	VISCOSITY	DAYS	VISCOSITY	DAYS
Sample 1, 6 RPM	0	1500	1	1500	7
Sample 1, 60 RPM	1442	1442	1442	1442	1442
Sample 2, 6 RPM	3300	3900	3800	3800	3800
Sample 2, 60 RPM	1990	2540	2590	2590	2590

20	25	20	25	20	25
STI	STI	STI	STI	STI	STI
Sample 1	1.04	1.04	1.04	1.04	1.04
Sample 2	1.66	1.53	1.53	1.53	1.51
pH					
5 Sample 1	11.33	11.33	11.33	11.33	11.33
Sample 2	11.29	11.41	11.41	11.41	10.99

FIG. 1 is a graph of the viscosity of a control sample (Sample 1), a waterborne epoxy resin without a rheology control agent, versus the present waterborne system (Sample 2) over time. Both Table 1 and FIG. 1 illustrate that the present waterborne system achieved stable 10 performance after about 1 day and a desired increase in viscosity.

EXAMPLE IV

A waterborne system, including an aqueous fumed silica dispersion (similar to the dispersion prepared in EXAMPLE I) and a waterborne epoxy resin, was prepared and evaluated to determine the effectiveness of the present dispersion as a thixotrope for waterborne systems. The aqueous fumed silica dispersion was mixed with EPI-REZ® 3510-W-60 15 nonionic, aqueous dispersion of bisphenol-A epoxy resin, available from Shell Chemical Co. Houston, TX, until a loading level of 2% dry silica on resin solids was achieved. The viscosity (centipoise), shear thinning index (STI), and pH were then measured after sample preparation (0 days), and after periods of 1 and 7 days. The viscosity was measured on a Brookfield viscometer using Nos. 3 or 4 spindle at 6 and 60 RPM's. The experimental results are presented below in Table 1.

20 TABLE 2

20 Using Nos. 3 or 4 spindle at 6 and 60 RPM's. The experimental results are presented below in 25 Table 2.

20	25	20	25	20	25
VISCOSITY	20	VISCOSITY	20	VISCOSITY	20
Sample 1, 6 RPM	1500	1500	1500	1500	1500
Sample 1, 60 RPM	1442	1442	1442	1442	1442
Sample 2, 6 RPM	3300	3900	3800	3800	3800
Sample 2, 60 RPM	1990	2540	2590	2590	2590

periods of 1 and 7 days. The viscosity was measured on a Brookfield viscometer using Nos. 3 or 4 spindle at 6 and 60 RPM's. The experimental results are presented below in Table 3.

TABLE 3

	0	1	7
Sample 1, 6 RPM	220	220	220
Sample 1, 60 RPM	178	178	178
Sample 2, 6 RPM	520	500	400
Sample 2, 60 RPM	264	270	260
STI			
Sample 1	1.24	1.24	1.24
Sample 2	1.97	1.85	1.85
pH			
Sample 1	3.41	3.41	3.41
Sample 2	5.70	7.04	7.04

FIG. 2 is a graph of the viscosity of a control sample (Sample 1), a waterborne bisphenol-A epoxy resin without a rheology control agent, versus the present waterborne system (Sample 2) over time. Both Table 2 and FIG. 2 illustrate that the present waterborne system achieved stable performance, particularly the Sample 2 measured at 60 RPM, and a desired increase in viscosity. The Sample 2 viscosity measured at 60 RPM decreased more rapidly.

EXAMPLE V

	0	1	7
Days			
Sample 1, 6 RPM	2700	2700	2700
Sample 1, 60 RPM	1010	1010	1010
Sample 2, 6 RPM	3000	5600	15400
Sample 2, 60 RPM	1130	1950	3700
STI			
Sample 1	2.67	2.67	2.67
Sample 2	2.65	2.87	4.16
pH			
Sample 1	8.74	8.74	8.74
Sample 2	9.38	9.36	7.40

A waterborne system, including an aqueous fumed silica dispersion (similar to the dispersion prepared in EXAMPLE I) and a waterborne epichlorohydrin and bisphenol-A epoxy resin, was prepared and evaluated to determine the effectiveness of the present dispersion as a thixotrope for waterborne systems. The aqueous fumed silica dispersion was mixed with EPI-REZ[®] 3510-WY-55 (55% solids) dispersion of Econ[™] 1001F condensation product of epichlorohydrin and bisphenol-A in water, available from Shell Chemical Co., Houston, TX, until a loading level of 2% dry silica on resin solids was achieved. The viscosity (centipoise), shear thinning index (STI), and pH were then measured after sample preparation (0 days), and after

A waterborne system, including an aqueous fumed silica dispersion (similar to the dispersion prepared in EXAMPLE 1) and a waterborne urethane modified epoxy resin, was prepared and evaluated to determine the effectiveness of the present dispersion as a thixotropic waterborne system. The aqueous fumed silica dispersion was mixed with EPI-REZ® 5520-60 nonionic aqueous dispersion of urethane modified epoxy resin, available from Shell Chemical Co., Houston, TX, until a loading level of 2% dry silica on resin solids was achieved. Viscosity (centipoise), shear thinning index (STI), and pH were then measured after sample preparation (0 days), and after periods of 1 and 7 days. The viscosity was measured on a Brookfield viscometer using Nos. 3 or 4 spindle at 6 and 60 RPM's. The experimental results are presented below in Table 4.

TABLE 4

TEST	TEST	TEST	TEST	TEST
1	2	3	4	5
6	7	8	9	10
11	12	13	14	15
16	17	18	19	20
21	22	23	24	25
26	27	28	29	30
31	32	33	34	35
36	37	38	39	40
41	42	43	44	45
46	47	48	49	50
51	52	53	54	55
56	57	58	59	60
61	62	63	64	65
66	67	68	69	70
71	72	73	74	75
76	77	78	79	80
81	82	83	84	85
86	87	88	89	90
91	92	93	94	95
96	97	98	99	100
101	102	103	104	105
106	107	108	109	110
111	112	113	114	115
116	117	118	119	120
121	122	123	124	125
126	127	128	129	130
131	132	133	134	135
136	137	138	139	140
141	142	143	144	145
146	147	148	149	150
151	152	153	154	155
156	157	158	159	160
161	162	163	164	165
166	167	168	169	170
171	172	173	174	175
176	177	178	179	180
181	182	183	184	185
186	187	188	189	190
191	192	193	194	195
196	197	198	199	200
201	202	203	204	205
206	207	208	209	210
211	212	213	214	215
216	217	218	219	220
221	222	223	224	225
226	227	228	229	230
231	232	233	234	235
236	237	238	239	240
241	242	243	244	245
246	247	248	249	250
251	252	253	254	255
256	257	258	259	260
261	262	263	264	265
266	267	268	269	270
271	272	273	274	275
276	277	278	279	280
281	282	283	284	285
286	287	288	289	290
291	292	293	294	295
296	297	298	299	300
301	302	303	304	305
306	307	308	309	310
311	312	313	314	315
316	317	318	319	320
321	322	323	324	325
326	327	328	329	330
331	332	333	334	335
336	337	338	339	340
341	342	343	344	345
346	347	348	349	350
351	352	353	354	355
356	357	358	359	360
361	362	363	364	365
366	367	368	369	370
371	372	373	374	375
376	377	378	379	380
381	382	383	384	385
386	387	388	389	390
391	392	393	394	395
396	397	398	399	400
401	402	403	404	405
406	407	408	409	410
411	412	413	414	415
416	417	418	419	420
421	422	423	424	425
426	427	428	429	430
431	432	433	434	435
436	437	438	439	440
441	442	443	444	445
446	447	448	449	450
451	452	453	454	455
456	457	458	459	460
461	462	463	464	465
466	467	468	469	470
471	472	473	474	475
476	477	478	479	480
481	482	483	484	485
486	487	488	489	490
491	492	493	494	495
496	497	498	499	500
501	502	503	504	505
506	507	508	509	510
511	512	513	514	515
516	517	518	519	520
521	522	523	524	525
526	527	528	529	530
531	532	533	534	535
536	537	538	539	540
541	542	543	544	545
546	547	548	549	550
551	552	553	554	555
556	557	558	559	560
561	562	563	564	565
566	567	568	569	570
571	572	573	574	575
576	577	578	579	580
581	582	583	584	585
586	587	588	589	590
591	592	593	594	595
596	597	598	599	600
601	602	603	604	605
606	607	608	609	610
611	612	613	614	615
616	617	618	619	620
621	622	623	624	625
626	627	628	629	630
631	632	633	634	635
636	637	638	639	640
641	642	643	644	645
646	647	648	649	650
651	652	653	654	655
656	657	658	659	660
661	662	663	664	665
666	667	668	669	670
671	672	673	674	675
676	677	678	679	680
681	682	683	684	685
686	687	688	689	690
691	692	693	694	695
696	697	698	699	700
701	702	703	704	705
706	707	708	709	710
711	712	713	714	715
716	717	718	719	720
721	722	723	724	725
726	727	728	729	730
731	732	733	734	735
736	737	738	739	740
741	742	743	744	745
746	747	748	749	750
751	752	753	754	755
756	757	758	759	760
761	762	763	764	765
766	767	768	769	770
771	772	773	774	775
776	777	778	779	780
781	782	783	784	785
786	787	788	789	790
791	792	793	794	795
796	797	798	799	800
801	802	803	804	805
806	807	808	809	810
811	812	813	814	815
816	817	818	819	820
821	822	823	824	825
826	827	828	829	830
831	832	833	834	835
836	837	838	839	840
841	842	843	844	845
846	847	848	849	850
851	852	853	854	855
856	857	858	859	860
861	862	863	864	865
866	867	868	869	870
871	872	873	874	875
876	877	878	879	880
881	882	883	884	885
886	887	888	889	890
891	892	893	894	895
896	897	898	899	900
901	902	903	904	905
906	907	908	909	910
911	912	913	914	915
916	917	918	919	920
921	922	923	924	925
926	927	928	929	930
931	932	933	934	935
936	937	938	939	940
941	942	943	944	945
946	947	948	949	950
951	952	953	954	955
956	957	958	959	960
961	962	963	964	965
966	967	968	969	970
971	972	973	974	975
976	977	978	979	980
981	982	983	984	985
986	987	988	989	990
991	992	993	994	995
996	997	998	999	1000

FIG. 4 is a graph of the viscosity of a control sample (Sample 1), a waterborne urethane modified epoxy resin without a rheology control agent, versus the present waterborne system (Sample 2) over time. Both Table 4 and FIG. 4 illustrate that the present waterborne system achieved stable performance after about 1 day and a desired increase in viscosity.

EXAMPLE VII

A waterborne system, including an aqueous fumed silica dispersion (similar to the dispersion prepared in EXAMPLE 1) and a waterborne acrylic copolymer resin, was prepared and evaluated to determine the effectiveness of the present dispersion as a thixotrope for waterborne systems. The aqueous fumed silica dispersion was mixed with Neocryl® A-639 waterborne acrylic copolymer, available from Zeneca Resins, Wilmington, MA, until a loading level of 2% dry silica on resin solids was achieved. The viscosity (centipoise), shear thinning index (STI), and pH were then measured after sample preparation (0 days), and after periods of 1 and 7 days. The viscosity was measured on a Brookfield viscometer using Nos. 3 or 4 spindle at 6 and 60

TABLE 5

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- 14 -

pH			
Sample 1	6.53	6.53	6.53
Sample 2	6.59	6.49	6.53

FIG. 5 is a graph of the viscosity of a control sample (Sample 1), a waterborne urethane modified epoxy resin without a rheology control agent, versus the present waterborne system (Sample 2) over time. Both Table 5 and FIG. 5 illustrate that the present waterborne system achieved stable performance after about 1 day and desired increase in viscosity.

EXAMPLE VIII

A coating formulation, incorporating the aqueous fumed silica dispersion of the present invention, a waterborne acrylic resin system, and various additives, was prepared and evaluated to determine the effectiveness of the present dispersion as a thixotrope in coatings. A high gloss waterborne topcoat, based on Neocryl® XK90 acrylic copolymer emulsion, available from Zeneca Resins, Wilmington, MA, was prepared. The coating formulation, as set forth below in Table 6, was prepared by first mixing the components of Part A in a Waring Blender at high speed for about 5 minutes. The Part B components were then added and dispersed into the Part A mixture in the Waring Blender at high speed for about 5 minutes. The viscosity (mPas), STI, and pH were then measured after periods of 2 days, 25 days, and 60 days. These measurements were taken for a high gloss aqueous top coat formulation without any commercial thickening agent (Control), and a similar formulation utilizing an aqueous fumed silica dispersion (similar to the dispersion prepared in Example 1) as an aqueous thixotrope (Sample). The viscosity of the coatings was measured on a Brookfield Rotary Viscometer RVD-II using spindle Nos. 1 or 2 at speeds of 0.5, 1.0, 2.5, 5.0, and 10.0 RPM. The STI is a ratio of the measured viscosity at 0.5 and 5.0 RPM's. The experimental results are presented below in Table 7.

TABLE 7

20 VISCOSITY (mPas)	DAYS		
	2	25	60
Control, 0.5 RPM	100	90	70
1.0 RPM	90	60	70
2.5 RPM	72	58	56
5.0 RPM	66	54	56
10.0 RPM	60	52	55
Sample, 0.5 RPM	2560	1280	1280
1.0 RPM	1720	1040	1080

25 FORMULATION	CONTROL (WT. %)	SAMPLE (WT. %)
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TABLE 6

2.5 RPM	1010	752	784
5.0 RPM	720	620	648
10.0 RPM	524	508	536
STI (0.5/5.0 RPM)			
Control	1.52	1.66	1.25
Sample	3.56	2.06	1.98

As illustrated above, the aqueous dispersion of the present invention can be incorporated not only in resins but also in coating formulations.

EXAMPLE IX

A coating formulation, incorporating the aqueous fumed silica dispersion of the present invention, a waterborne alkyd resin system, and various additives, was prepared and evaluated to determine the effectiveness of the present dispersion as a thixotrope in coatings. An aqueous industrial maintenance coating based on Resydrol® AY 466 W, 38% alkyd resin emulsion, available from Hoechst Celanese, Fine Chemicals Division; Charlotte, NC, was prepared. The coating formulation, as set forth below in Table 8, was prepared by mixing the components in a Waring blender at high speed for about 5 minutes. The viscosity (mPas) and STI were then measured after periods of 1, 18 and 60 days. These measurements were taken for an aqueous industrial maintenance coating of formulation with and without a commercial thickening agent (Borchiegel™ L75N, 54%), and a similar formulation utilizing an aqueous fumed silica dispersion (similar to the dispersion prepared in Example 1) as an aqueous thixotrope (Sample). The viscosity of the coatings was measured on a Brookfield Rotary Viscometer. The experimental results are presented below in Table 9. FIG. 6 is a graph illustrating the viscosity aging over time, taken from the data in Table 9, and corresponding to a shear rate of 1.4 dynes/sec. At a finesness of grind <10 microns (the graph was converted from mPas to centipoise). As illustrated in FIG. 6, the aqueous fumed silica dispersion achieved and maintained a stable workable viscosity for an extended period of time. The Control sample exhibited a high initial increase in viscosity and substantial loss over time.

TABLE 8

FORMULATION	CONTROL (WT. %)	SAMPLE (WT. %)
Resydrol® AY466 W, 38%	70.00	69.02
Aqueous Ammonia, 10%	3.00	2.96
Additol™ VXXW 4940 drying agent (Hoechst Celanese)	1.30	1.28
Titanox™ 2300 (Kronos)	21.24	20.94
Borchigel™ L7SN, 54% thickener	1.86	—
Aqueous Fumed Silica Dispersion (i.e., Example 1)	—	4.81
Additol™ VXXW 4973 defoamer	0.30	0.30
Surfynol™ SE-F wetting agent (Air Products)	0.30	0.30
Additol™ XL 297 antiskinning agent	0.40	0.39
Deionized water	1.60	—
	100.00	100.00

TABLE 9

STI	Sample	1.985	2.940	3.087
20	Control	6.660	4.340	3.472
25	Control without Borchigel™ thickener	0.785	0.588	0.588
60				

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- 18 -

Control	1.54	1.27	1.66
Control without thickener	3.13	1.81	1.88
Sample	3.33	4.64	2.63

CLAIMS

As illustrated above, the aqueous dispersion of the present invention can be incorporated not only in resins but also in coating formulations.

Although particular embodiments of the invention have been described in detail for purposes of illustration, various changes and modifications may be made without departing from the scope and spirit of the invention. Accordingly, the invention is not to be limited except as by the appended claims.

What is claimed is:

1. A waterborne system, comprising:
an aqueous dispersion of fumed silica; and
a waterborne resin;
2. The waterborne system of claim 1, wherein said fumed silica has a surface area between about 85 m²/g and about 410 m²/g.
3. The waterborne system of claim 2, wherein said fumed silica has a surface area of about 200 m²/g.
4. The waterborne system of claim 1, wherein said fumed silica has an impurity level of less than 1%.
5. The waterborne system of claim 1, wherein said system comprises between about 0.5 and about 10.0% fumed silica, by weight, of total resin solids.
6. The waterborne system of claim 5, wherein said system comprises between about 0.5 and about 5.0% fumed silica, by weight, of total resin solids.
7. The waterborne system of claim 6, wherein said system comprises about 2.0% fumed silica, by weight, of total resin solids.
8. The waterborne system of claim 1, wherein said aqueous dispersion of fumed silica has between 10% and 45%, by weight, silica solids.
9. The waterborne system of claim 8, wherein said aqueous dispersion of fumed silica has between 15% and 30%, by weight, silica solids.

10. The waterborne system of claim 9, wherein said aqueous dispersion of fumed silica has 20%, by weight, silica solids.

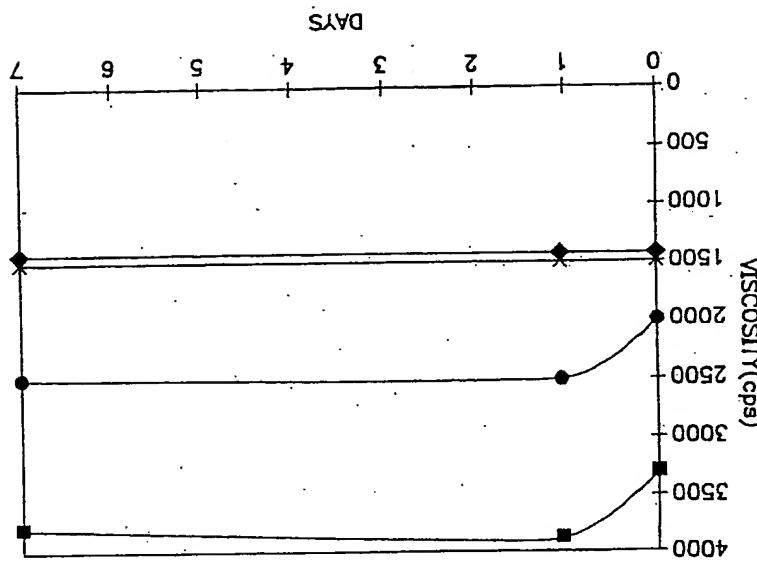
11. The waterborne system of claim 1, wherein said aqueous dispersion of fumed silica has a pH between about 5.0 and about 10.5.

5 12. The waterborne system of claim 11, wherein said aqueous dispersion of fumed silica has a pH between about 7.0 and about 9.5.

13. The waterborne system of claim 1, further comprising at least one additive selected from the group consisting of surfactants, coalescing solvents, pigments, defoamers, antiskinning agents, drying agents, wetting agents, thickening agents, dispersing agents, 10 biocides, and corrosion inhibitors.

14. The waterborne system of claim 1, wherein said waterborne resin is selected from the group consisting of alkyd, acrylic, polyester, polyether, silicate, urethane, epoxy, formaldehyde, vinyl, and mixtures thereof.

● - SAMPLE 2,60 RPM
■ - SAMPLE 2,6 RPM
◆ - SAMPLE 1,60 RPM
* - SAMPLE 1,6 RPM



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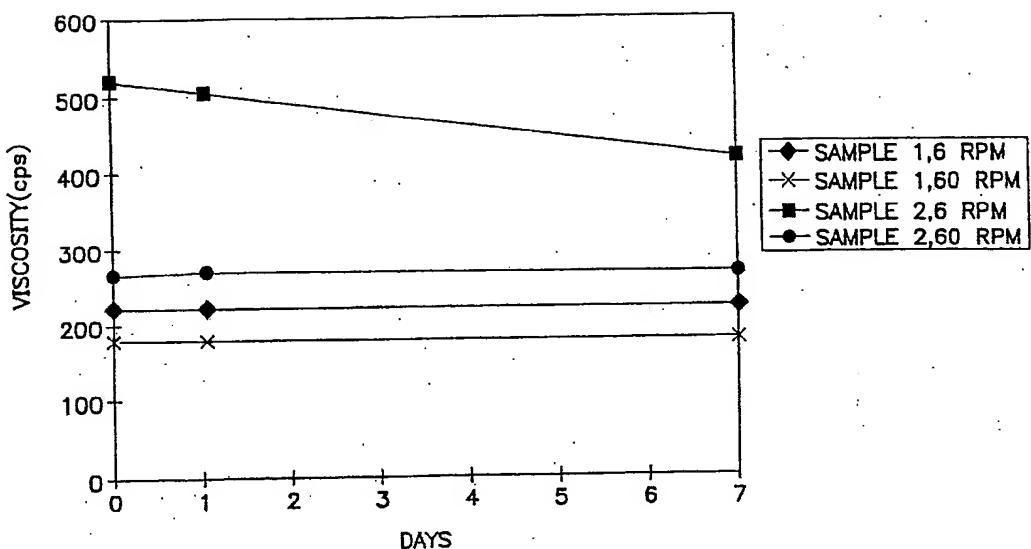


FIG. 2

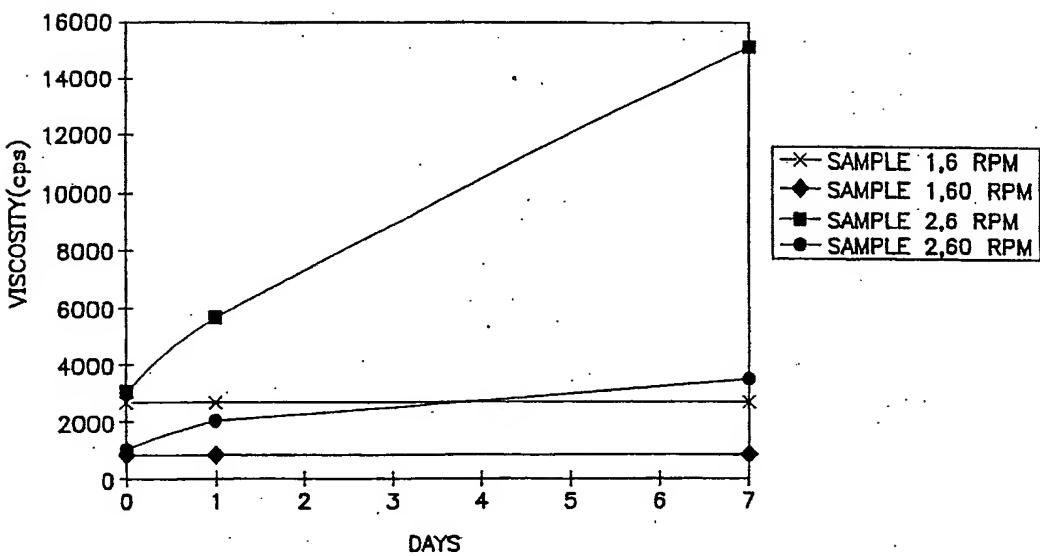


FIG. 3

FIG. 4

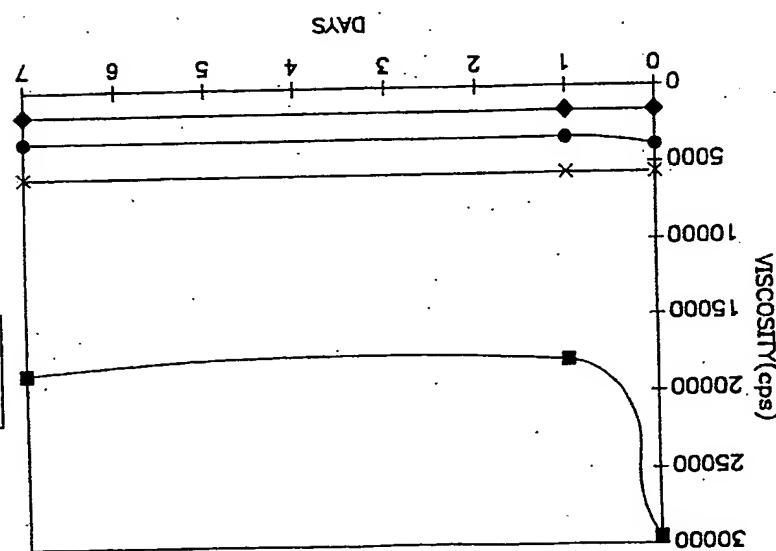
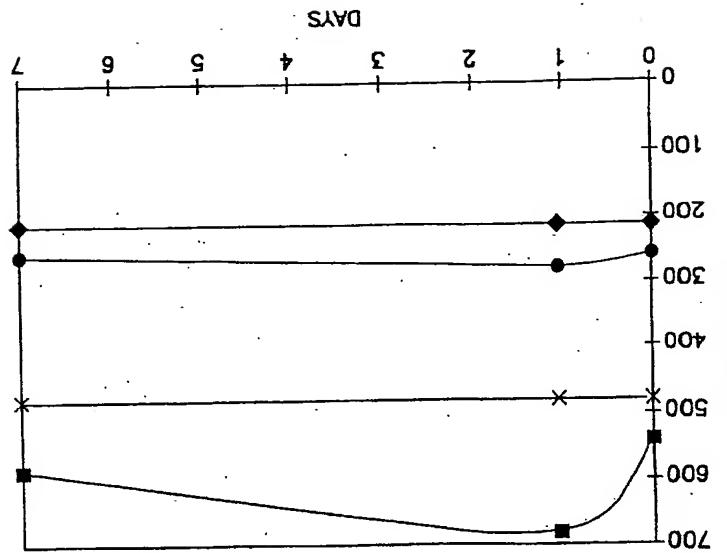


FIG. 5



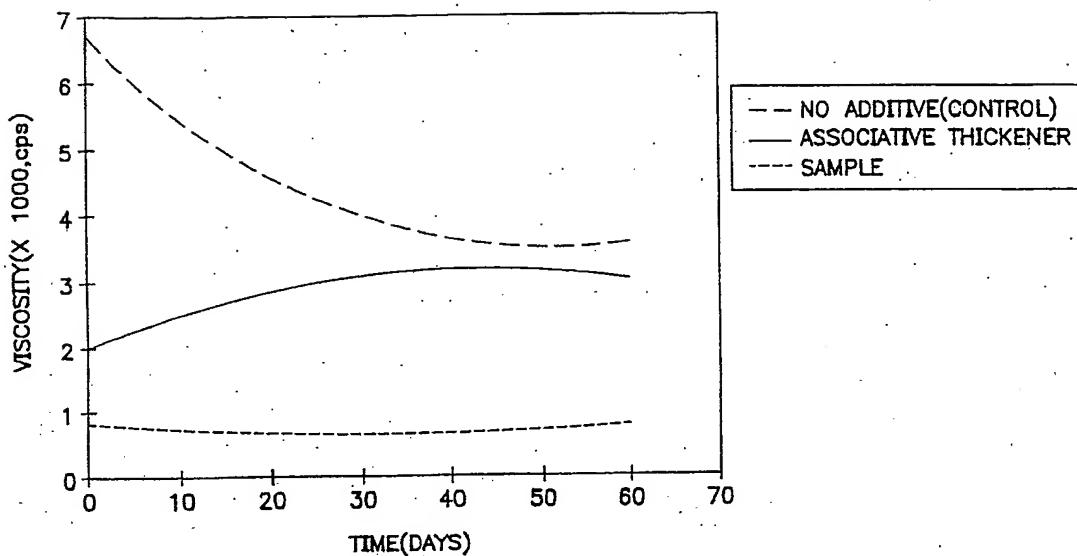


FIG. 6

INTERNATIONAL SEARCH REPORT

One "World Application No.
PCT/US 96/15940

▲ CLASSIFICATION OF SUBJECT MATTER
IPC 6 C09D5/04

According to International Patent Classification (IPC) or to both National classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 C09D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category Classification of documents, with indication, where appropriate, of the relevant passages

Reference to claim No.

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November 1990 14
see abstract
see page 2, line 48-52
see page 3, line 1-5
see page 4, paragraph 3
A WO 94 18277 A (CARBOT CORPORATION) 18 1
August 1994
see abstract
see claim 2
A US 4 455 331 A (ROBERT J. BARSOOTTI) 19
June 1984
see claims 1-3

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Parent Family members are listed in annex.

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2

Date of the actual completion of the international search
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Date of making of the international search report
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INTERNATIONAL SEARCH REPORT

Information on patent family members

		Information on patent family members		Int'l. Search Application No PCT/US 96/15940	
Patent document cited in search report	Publication date	Parent family member(s)		Publication date	
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